

BEST AVAILABLE COPY

PL 1155782

THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office

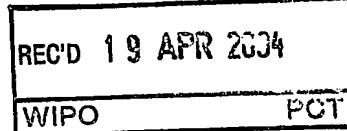
April 15, 2004

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM
THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK
OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT
APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A
FILING DATE.

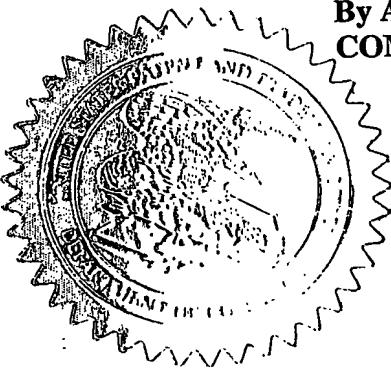
APPLICATION NUMBER: 60/445,607

FILING DATE: *February 07, 2003*

RELATED PCT APPLICATION NUMBER: PCT/US04/03572



By Authority of the
COMMISSIONER OF PATENTS AND TRADEMARKS


N. WOODSON
Certifying Officer

PRIORITY
DOCUMENT

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)



02-10-03 601445607 02/07/03

Docket Number 3212B

A/for
S

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
PROVISIONAL APPLICATION COVER SHEET

Box Provisional Patent Application
Commissioner for Patents
Washington, DC 20231

JC912 U.S. PTO
601445607
02/07/03

(A) The attached document is filed as a Provisional Application for patent under 37 CFR 1.53 (c)(1).

(B) Inventors (*including* city and state or foreign country of residence, for each)
or alphanumeric identifier:

Saurabh S. Lawate, 10060 Stonehollow Road, Concord, Ohio 44060

Robert Silverstein, 4 Park Place, Great Neck, New Jersey 11024

(C) Title: Gel Composition for Optical Fiber Cable

(D) Attorney/Agent: Samuel B. Laferty Registration No. 31,537

(E) Docket Number: 3212B

(F) Send Correspondence to: Docket Clerk
Patent Department/Mail Drop 022B
The Lubrizol Corporation
29400 Lakeland Blvd.
Wickliffe, Ohio 44092-2298

(G) Fees: Charge the fee for filing of a provisional application, believed to be \$160.00, to Deposit Account No. 12-2275 (The Lubrizol Corporation). Any deficiency or overpayment in fees should be charged or credited to the same account.

(H) 7 Pages of Specification
0 Sheets of Drawings

3 Pages of Claims
1 Pages of Abstract

(I) A duplicate of this Cover Sheet is attached.

Registration No. 31,537
Telephone: 440-347-1541
Facsimile: 440-347-1110

Samuel B. Laferty
Attorney for Applicant

Samuel B. Laferty
Signature

February 7, 2003
Date

I certify that this correspondence is being deposited with the United States Postal Service as Express Mail No. EU381349110US in an envelope addressed to: Box Provisional Application, Commissioner for Patents, Washington, DC 20231, on: February 7, 2003.

By: Nancy S. Dedeck
Nancy S. Dedeck

Date: 2-7-03

3212B

Title: Gel Composition for Optical Fiber CableFIELD OF INVENTION

5 Composition of optical fiber gels having compatibility with polymeric sheathings commonly used in optical fiber cables. The gels in filled cables minimize the intrusion of water and other harmful compounds into filled information transmission cables such as optical fiber cables. The gels along 10 with the cable sheath protect the internal wires, fibers etc. from stresses applied to the cables sheath.

BACKGROUND OF THE INVENTION

15 The optical fiber cable industry manufactures optical fiber cables by encasing the optical fibers in a polymeric sheathing. A jelly is placed between the polymeric sheathing and the optical fiber. The purpose of this jelly is to provide water resistance and as a buffer to bending stresses and strains.

20 Typical sheathing materials are polymeric in nature with polypropylene (PP) and polybutylterephthalate (PBT) being the most commonly used sheathing materials.

25 The jelly is usually a non-Newtonian oil. The non-Newtonian nature allows the jelly to thin out during processing and set after the processing shear forces are removed. Critical parameters that impart the necessary performance are viscosity at various shear rates and the yield stress. Typically the jelly is made using oil and an inorganic or organic thickener. Inorganic thickeners used range from organic clays to silica. These thickeners are suspended in a hydrophobic oil such as a mineral oil or synthetic oil. Additionally, stabilizers may be incorporated to ensure oxidative stability of the mixture.

30 The oil chosen has a profound influence on the compatibility of the jelly with the sheathing material. Typically, mineral oil based jellies are compatible with PBT sheathing whereas synthetic hydrocarbon oil based jellies are compatible with PP sheathing materials. Synthetic jellies are more expensive than mineral oil based jellies and there is a need for cheaper jellies that will be

compatible with PP sheathings. Moreover, there is a need for a cheaper jelly that would be compatible with both PP and PBT sheathings i.e. a 'universal product'.

Very few examples exist in prior art which identify jelly compositions that are compatible with both sheathing materials. As an example US Patent 5 5672640 outlines the use of castor oil and a ricinoleate polyol with colloidal particles. US Patent 5672640 also highlights the critical problem that expensive components have to be used in order for the jelly to be compatible with PP sheathing materials. US Patent 5672640 clearly outlines the need for 10 low cost cable filling compounds that are compatible with PP and provides a solution for the problem via the use of castor oil derivatives. Unfortunately, to be useful there is a need for high loading of silica in these formulations which adds cost.

15 US4701016 outlines the use of various mineral and synthetic base oils but the drawback is that it uses very high loadings of silica. This can add to cost very significantly.

20 US5905833 discusses the use of a jelly composition containing mineral oils and a thickening system. The thickening system contains silica's and a polymer. High molecular weight polymers are used. The primary drawback of this is that the polymer itself is expensive and requires very long processing times in order to solubilize it into the base oil used. It is thus desirable to eliminate the use of such polymers.

SUMMARY OF THE INVENTION

25 The present application solves the problem of non-compatibility with polypropylene of certain filling compositions widely used in the industry without sacrificing desired performance or increasing cost. The use of a blend of polydecene and polybutene in ratios as outlined in the preferred embodiments of the present invention enables that the jelly is compatible with 30 PP and PBT without sacrificing low temperature performance. Also disclosed are optimized compositions for gels for fiber optic cables derived from oil, colloidal silica filler, a high molecular weight polymer and optional functional additives. Gel compositions were developed based on the blend of polydecene

and polybutene basestocks and thickeners, which are compatible with the PP and PBT sheathings (e.g. they do not soften or deteriorate the sheath material).

Further, the preferred formulations have eliminated the use of very high loadings of silica as outlined in US4701016 or the use of high molecular weight polymers as in US5905833. This has been accomplished using a higher surface area silica gel and a polyglycol coupling agent. Use of this combination enables the use of lower loadings of silica without the use of polymeric thickeners.

Other objects and advantages of the present invention will become apparent to those skilled in the art from the following detailed description read in conjunction with the claims appended hereto

DETAILED DESCRIPTION

The gel composition generally comprises a base oil, a high molecular weight polymer, a colloidal silica, and optionally coupling agents and antioxidants.

Base Oil

The base oil can be any of the American Petroleum Institute's (API) Group IV, or Group V basestock. Typical base oils include PAOs, polyglycol, polydecenes, and polybutenes. Specific examples of this type of component include polyalpha olefin and other synthetic oils such as polyglycol and polybutene. The amounts of base oil in the compositions of the present invention are generally from about 80 to about 97 weight percent and more desirably from about 86 to about 96.

Colloidal Particulate

Colloidal hydrophobic and hydrophilic silica used individually or in combination. The colloidal particulate can be hydrophobic and or hydrophilic fumed silica or other particles such as iron and other inorganic particulate materials. Specific examples of this type of component include Aerosil and Cabosil silicas from DeGussa and Cabot corporations. The amounts of colloidal particulate in the compositions of the present invention are desirably from about 1 to 50 weight percent, more desirably from about 2 to 10 weight percent, and preferably from about 4 to about 8 weight percent.

The colloidal particulate provides a particular type of viscosity modification to the gel not available from high molecular weight soluble polymers. When sufficient colloidal material is present, the surfaces of adjacent particulate materials can hydrogen bond to adjacent particles forming a network that is resistant to stress. This provides thixotropic behavior, high yield stress values, and bleed resistance (anti-drip). Above a certain stress value these hydrogen bonds are broken and the gel deforms without memory of its previous shape and the hydrogen bonds between adjacent particles reform to re-establish a rigid network.

10 **Coupling Agent(s)**

Coupling agents are optional and function to couple the particulate material into a more continuous network building viscosity or modulus without adding more particulate material. Coupling agents generally are capable of hydrogen bonding with hydroxyl groups on the colloidal particulate material. 15 Coupling agents with hydroxyl groups are preferred (e.g. monofunctional and polyfunctional alcohols). They can be monomeric, oligomeric, or polymeric. Specific examples of this type of component include polyglycols (including but not limited to poly(alkylene oxide) and other polyols).

20 The amounts of coupling agents are generally up to 2 or 5 weight percent, more desirably from about 0.1 to about 2, and preferably from about 0.1 to about 0.5, and preferably from about 0.1 to about 0.3 weight percent.

Other Optional Additives

25 Other additives include antioxidants, hydrogen absorbing agents, surfactants, antiwear (including EP) agents, and antifoam agents. These may or may not be necessary depending upon the particular application of the gel and transmission cable. The antioxidants help increase oxidative induction time, ameliorate changes in the molecular weight of the oil and high molecular weight polymer, and reduce adverse color changes in the gel. Without them, depending on the resistance of the oil and polymer to oxidation, the oil and 30 polymer might degrade into lower molecular weight components (possibly volatile), or higher molecular weight components (possibly sludge), and or a combination of lower and higher molecular weights (generating both more

volatility and more sludge). The antifoam agents would help reduce the inclusion of gas bubbles in the gel and reduce foaming above the surface of the gel.

5 The amounts of optional functional components in the compositions of the present invention are generally up to 5 weight percent, more desirably from about 0.1 to about 1, and preferably from about 0.1 to about 0.5 weight percent. The particular relationship between the amounts and types of the above components is by weight.

Recipe I: Synthetic Oil Based Recipe

10

Ingredient	Manufacturer	Quantity used for a 10 gallon batch (lbs)	Wt. Percent
PAO-40 Mobil SHF4 (base oil)	Mobil	4.567	45.67%
Polybutene H100	Chemcentral	2.855	28.55%
Polybutene H300	Ciba	2.093	20.93%
Irganox L135 (antioxidant)	Ciba Gigy	0.045	0.45%
Aerosil 300VS - Hydrophilic Silica	Degussa	0.400	4.00%
Polyglycol 2000 (coupling agent)	Dow Chemical	0.041	0.41%
Total		10.00	100%

The gels can be prepared by any method that disperses the silica and the other components uniformly in the oils. Such procedures are disclosed in the prior art. A preferred procedure is listed below.

Procedure

15 • Mix the H100, H300 and SHF4 in a beaker on a hot plate at about 70-80C using a spatula

- Transfer the mixed base oils to the 2 gallon unit and maintain at 75F (Stirring: 25 rpm anchor; 6800 rpm disperser; 5500 rpm emulsifier).
- Mix for 5 minutes and turn off disperser and emulsifier (to prevent possible shearing of polybutene). Turn on Mokon heating unit to 110F. Batch

20

temp is about 110 F. Stir for 15 minutes. (Stirring; Anchor 50 rpm; disperse and emulsifier 0 rpm).

- Pull a sample out for ASTMD445 viscosity at 100 C
- Sample looks white and frothy at this stage with a lot of air entrapped
- 5 • Put Irganox L135 and Aerosil 300 VS and mix for 5 mins (Stirring: 22 rpm anchor; 4050 rpm disperser; 4500 rpm emulsifier). Temp is about 110F due to mixing.
- Stop stirring, lift mixer and clean mixer area and lid to remove solid silica.
- 10 • Continue mixing for 10 mins (80 rpm anchor; 6450 rpm disperser; 0 rpm emulsifier)
- Add polyglycol and mix 30 min ((80 rpm anchor; 6450 rpm disperser; 4500 rpm emulsifier).
- Turn off stirring except anchor at 40 rpm. Pull vacuum till batch is
- 15 clear. Note that in this case the vacuum was weak so the mixture was heated to about 130 F after removing about ½ the quantity of material. It took about 1 hr to get product with no air entrapped. (Note: when system is under vacuum there are always bubbles at the top layer. Only way to check deareation is by stopping the vacuum/mixing and looking at sample).

20 While the invention has been explained in relation to its preferred embodiments, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to be understood that the invention disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

25 Each of the documents referred to above is incorporated herein by reference. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." Unless otherwise indicated, 30 each chemical or composition referred to herein should be interpreted as being a commercial grade material which may contain the isomers, by-products,

derivatives, and other such materials which are normally understood to be present in the commercial grade. However, the amount of each chemical component is presented exclusive of any solvent or diluent oil, which may be customarily present in the commercial material, unless otherwise indicated. It
5 is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. While ranges are given for most of the elements of the invention independent of the ranges for other elements, it is anticipated that in more preferred embodiments of the invention, the elements of the invention are to be combined with the various (assorted)
10 desired or preferred ranges for each element of the invention in various combinations.

As used herein, the expression "consisting essentially of" permits the inclusion of substances that do not materially affect the basic and novel characteristics of the composition under consideration. Comprising means
15 having at least the listed elements and optionally a variety of other unnamed elements that might affect the basic characteristics of the composition.

3212B

What is claimed is

- 5 1. A non-newtonian jelly composition suitable for use in optical fiber cable manufacture comprising:
 - a. a synthetic oil of lubricating viscosity,
 - b. a fumed silica,
 - c. an antioxidant, and
 - 10 d. a coupling agent.
2. The composition of claim 1 wherein the fumed silica is hydrophobic or hydrophilic.
- 15 3. The composition of claim 1 wherein the fumed silica is hydrophilic and the composition is substantially free of hydrophobic silica, less than 0.1%.
4. The composition of claim 1 wherein the fumed silica has an average primary particle size ranging from 5-30 nm.
- 20 5. The composition of claim 1 wherein the fumed silica has an average specific BET surface of 150-400 m²/g.
6. The lubricating composition of claim 1 wherein the fumed silica is present from 1 to 10% by weight.
- 25 7. The composition of claim 1 wherein the fumed silica is present from 3-6% by weight.
8. The composition of claim 1 wherein the preferred synthetic oil is chosen from a group of polydecenes, polyisoprenes, polyisobutenes, polybutenes
9. The composition of claim 1 wherein the synthetic oil comprises at least 85% of the composition by weight.
- 20 10. The composition of claim 1 wherein the synthetic oil is a mixture of at least two oils chosen from the group of polydecenes and polybutenes.
11. The composition of claim 1 wherein the coupling agent is a chemical with at least one hydrogen bonding site.
- 30 12. The composition of claim 1 wherein the coupling agent is a polyglycol.

13. The composition of claim 12 wherein the polyglycol has an average MW of at least 1000.
14. The composition on claim 1 comprising an antioxidant
15. The lubricant composition of claim 1 wherein the antioxidant is a hindered phenol antioxidant.
5
16. The lubricant composition of claim 16 wherein the antioxidant is present from 0.1 to 2% by weight.
17. The composition of claim 1 wherein the synthetic oils are a mixture of polydecene and polybutene, the silica is hydrophilic and the coupling agent is a polyglycol.
10
18. The composition of claim 19 where the polybutene has an average MW of less than 2000.
19. The composition of claim 19 wherein the preferred amount of polybutene used is at least 40% by weight of the total formulation.
15
20. The composition of claim 19 wherein the polyglycol has a molecular weight of 2000.
21. The composition of claim 19 where the preferred composition comprises a mixture of polydecene and polybutene in a ratio of 1:1
22. The composition of claim 19 wherein the preferred composition comprises a hydrophilic silica with a surface area of at least 150 m²/g
20
23. The composition of claim 19 wherein the hydrophilic silica has a surface area of greater than 250 m²/g
24. The composition of claim 19 wherein the mixture of synthetic oils comprises polydecenes and polybutenes
25. The composition of claim 19 wherein the coupling agent comprises at least 0.3% of the formulation
26. The composition of claim 19 which optionally comprises an antioxidant.
27. The composition of claim 28 wherein the antioxidant is a hindered phenol
30
28. The composition of claim 28 wherein the antioxidant is an amine
29. The composition of claim 28 wherein the antioxidant is selected from a group of antioxidants comprising phenolic and amine antioxidants.

30. The composition of claim 1 wherein the preferred composition comprises a blend of synthetic oils and a blend of fumed silicas.
31. The composition of claim 32 wherein the blend of silicas comprises hydrophobic and hydrophilic silicas.
- 5 32. The composition of claim 1 optionally comprising a high molecular weight polymer.
33. The composition of claim 1 wherein the high molecular weight polymer is a styrene butadiene polymer.

3212B

ABSTRACT

A gel composition from a synthetic lubricating oil, fumed silica, an antioxidant and a coupling agent for the fumed silica is described. The
5 coupling agent provides unique properties to the gel.

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.